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Synthetic Organic Chemicals

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A Fractionating Vacuum Pump and Some New Pump Oils

THE vacuum of the well-known Langmuir condensation pump is limited by the vapor pressure of the operating fluid. For many years, mercury was the only fluid available, and a vacuum higher than 10^{-3} mm. could not be secured unless a refrigerated trap was attached to the pump.

During the past few years a number of organic liquids of exceedingly low vapor pressure have been recommended for use in condensation pumps. Although they provide faster pumping than mercury, they do not operate against as high a backing pressure and they present certain inherent shortcomings. They dissolve air and retain semi-volatile substances which spoil a high vacuum, and they may show thermal decomposition or reaction with incoming vapors. They are difficult to secure or preserve in a condition of sufficient purity to yield their highest vacuums, as predicted from the known temperature-vapor pressure relation.

Recently, fractionating pumps have been designed in the laboratories of the Eastman Kodak Company which enable the purification to be accomplished or preserved during the act of pumping, even though the pump is handling reactive or partly condensible vapors. These pumps permit the use of liquids of exceedingly low vapor pressure which have hitherto been considered unsuitable, due to the possibility of thermal decomposition.

The vacuum in a simple, oil-operated condensation pump is impaired by:

(1) Air dissolved by the oil on the high-pressure, output side of the pump.

(2) Semi-volatiles dissolving in the oil.

(3) The presence of impurities which decompose thermally.

(4) The presence of non-volatile constituents which impede the evaporation of the true operating fluid and thus induce decomposition.

Factor 2 is of first importance; factors 1, 3, and 4 are secondary. The simple vertical pump, Figure 1, page 2, takes care of the semi-volatiles by rejecting them continuously into the fractionating lobes. The size illustrated, which is made of 1½″ tubing, requires a backing pressure of less than .08 mm. and has a speed at 10^{-3} mm. of 1 to 1.5 liters per second. The minimum pressure obtainable, irrespective of the nature of the filling, is dependent on the perfection of the backing vacuum. When this is about .08 mm., the best vacuum is about 10^{-4} mm., as measured by the ionization gauge. The pump is suitable for exhausting small molecular stills and should find routine use in organic or biological laboratories.

All four factors are taken care of in a "3-boiler" pump which really provides four active storage regions—A, B, C, and D, Figure 2. The pump can take a variety of forms, but that shown serves well in laboratory use. The pump requires a backing pressure of .12 mm. Hg, or less, and has a speed of 10 liters per second at 10^{-3} mm. The highest vacuum attainable has proved to be about 5×10^{-8} mm. at 20° C., using n-octyl phthalate. The pump need not be water-cooled. It operates well in the draught from a small fan or from two compressed air jets (Bunsen burners are economical jets), one directed at each of the working nozzles.

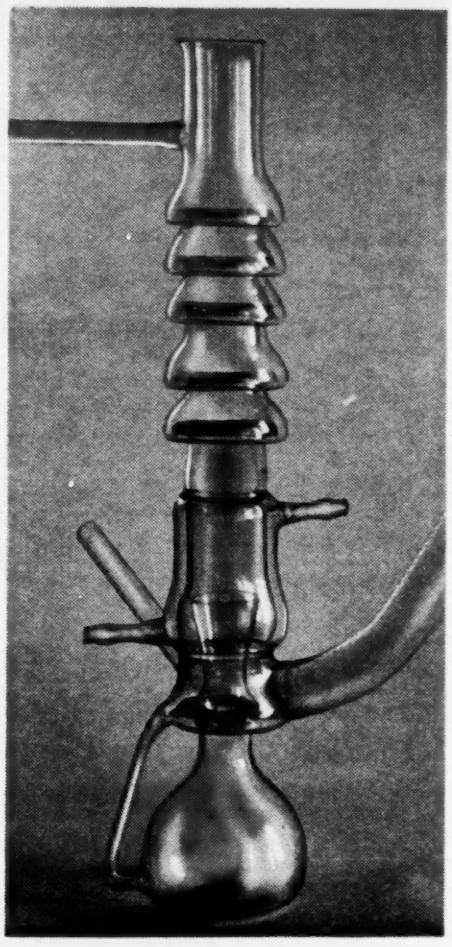

FIGURE 1

The cycle of operation is as follows: The oil vaporizes in the three compartments, indicated as A, C, and D, Figure 2. Vapor ascends from A and rejects air to the mechanical pump and volatiles to the catchment lobes (B); it also induces a moderate vacuum in the high vacuum receiving jet (E). The liquid in C vaporizes, causing pumping at the Crawford jet (F) and condenses in E, but the condensate can only return to C by way of A where it is boiled and robbed of lighter constituents. All the non-volatile impurities would eventually accumulate in C were it not for compartment D to which they pass to make up losses from evaporation. Thus, after a short period of operation, the volatiles are segregated in B, the tarry and colored residues in D, while the pure pump fluid boils in C to yield its optimum vacuum. Each of the boilers should be fitted with small electric heaters, Figure 3, which can be made conveniently by winding nichrome wire around split circles of mica. If the heaters are to be connected in series, they should be wound with the following relative lengths of similar wire A : C : D :: 4 : 3 : 1. The over-all resistance should be about

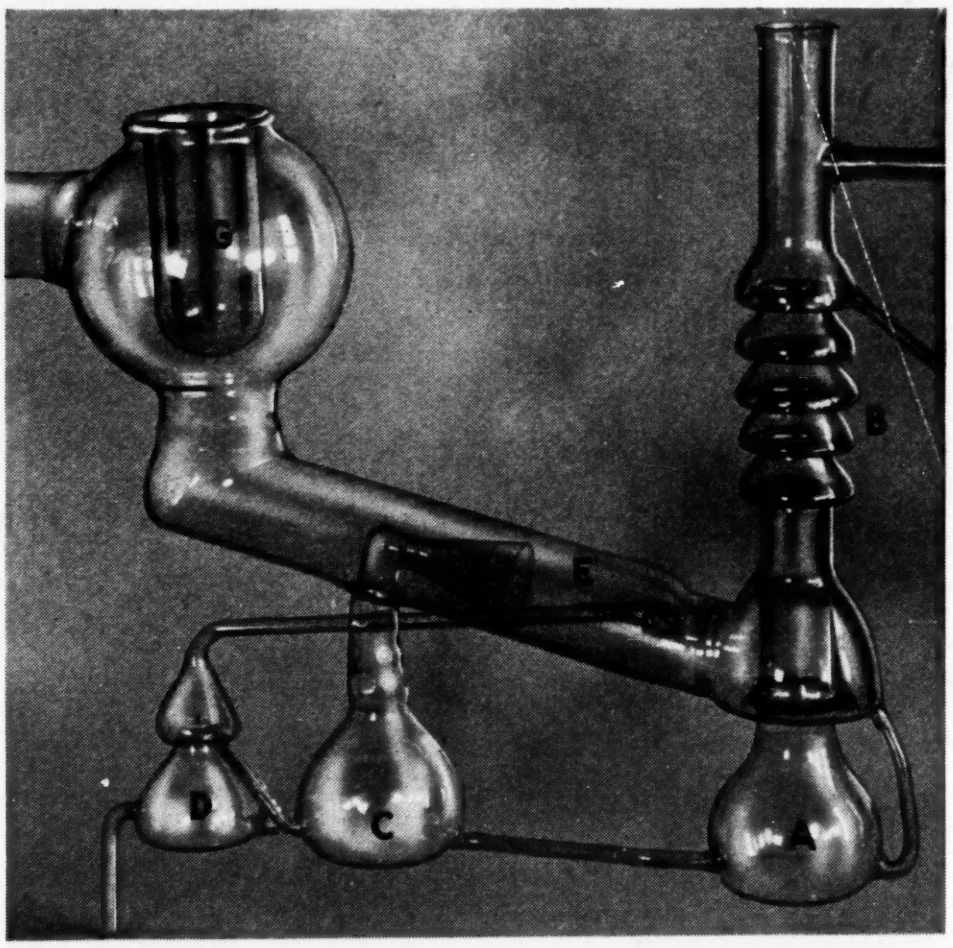

FIGURE 2

Dimensions

A—Jet clearance 3 mm.

E—2" diameter

F—3 mm. clearance between E and F.

50 ohms for operation from 110-volt mains and a rheostat 0-50 ohms should be placed in the circuit to accommodate different kinds of pump fluid and variations in room temperature and means of cooling. The boilers should be lagged with asbestos rope or wool felt.

Of many liquids examined in the Eastman laboratories, the esters of phthalic acid have been found best for fractionation pumps. The most important compounds are listed in heavy type in the accompanying table. These oils have been used in the laboratory continuously for many months without any signs of decomposition.

The most robust high vacuum oil is heptyl phthalate, but the most practical on a price basis are the n-amyl and ethyl hexyl phthalates. For convenience, the high vacuum grades of these liquids have been named *Amoil* and *Octoil*, respectively. Amoil is recommended for distillation and chemical work, and Octoil for physical use, such as x-ray, lamp, neon sign, spectrographic, mirror splluttering, metal evaporation, etc.

In using the phthalates, no charcoal trap is necessary, while phosphorous pentoxide is definitely harmful. A bent tube, baffle, or ice trap should be interposed between pump and apparatus to intercept the stream of oil vapor which passes back from the jet. Even when this has been attended to, oil is liable to deposit on distant parts of the evacuated apparatus. It is wise to provide a cold "sink" to reduce the pressure of the oil vapor below the saturation point. The sink need be only a few degrees cooler than room temperature and may be anywhere in the apparatus, though it is most convenient when situated at the pump entrance as shown at G, Figure 2. The function of this cold point is not to be confused with that of the usual trap filled with solid CO_2 or liquid air.

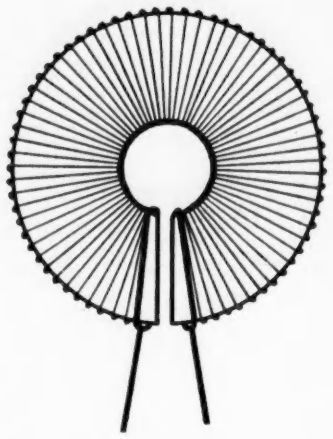

FIGURE 3

A detailed account of the pumps and oils will appear shortly in an article by K. C. D. Hickman, in the *Journal of the Franklin Institute.*

Name	*Eastman Cat. No.*	*B.P. at 1 mm.*	*Lowest pressure experimentally attained* at 25°*	*Lowest pressure experimentally attained* with a trap cooled by ice*
n-Butyl Phthalate	1403 X	149	2.0×10^{-4}	2.0×10^{-6}
n-Amyl " (Amoil)	2009 X	164	2.5×10^{-5}	5.0×10^{-7}
n-Hexyl "	4098 X	179	3.0×10^{-6}	5.0×10^{-7}
n-Heptyl "	4100 X	195	2.5×10^{-7}	1.2×10^{-7}
n-Octyl "		211	5.0×10^{-8}	5.0×10^{-8}
2-Ethyl Hexyl " (Octoil)**	4099 X	184 Approx.	5.0×10^{-7}	5.0×10^{-8}
Benzyl Phthalate†	2485 X	224	1.2×10^{-7}	1.0×10^{-7}

*In a 3-compartment pump and measured by ionization gauge. The "molecular" pressures are about ten times less.

**A mixed ethyl hexyl phthalate.

†Evinces traces of decomposition and solidifies in pumps on long standing unused.

The National Symposium of Organic Chemistry

ON THE tenth anniversary of its founding, the National Symposium of Organic Chemistry will convene in its "birthplace," Rochester, N. Y., December 30, 1935.

This year's program may be expected to measure up to the high standards set in the past. The usual custom of scheduling only a limited number of papers has been followed in order that the meetings may not be hurried and that ample time may be available for discussion.

The following outstanding members of the chemical profession will present papers at the symposium this year:

Homer Adkins, University of Wisconsin
Marston T. Bogert, Columbia University
Benjamin T. Brooks, New York City
Wallace H. Carothers, E. I. DuPont de Nemours
James B. Conant, Harvard University
Vincent du Vigneaud, George Washington University
Louis F. Fieser, Harvard University
Henry Gilman, Iowa State College
Arthur J. Hill, Harvard University
John R. Johnson, Cornell University
Morris S. Kharasch, University of Chicago
C. Frederick Koelsch, University of Minnesota
Carl S. Marvel, University of Illinois
Lyndon F. Small, University of Virginia
George H. Whipple, University of Rochester
M. L. Wolfrom, Ohio State University

A complete list of the titles of papers to be presented by the above speakers is not as yet available, but the program is to be composed of well-rounded discussions of the particular fields in which the speakers specialize. Summarizing reviews of the status of special lines of investigation will be given, including the speaker's own work, with some indications of the direction which future investigations may take.

Sessions will be held morning and afternoon Monday, Tuesday, and Wednesday, December 30 and 31, 1935 and January 1, 1936. There will be two evening meetings also: Monday evening the Symposium will be addressed by Dean George H. Whipple of the University of Rochester Medical School, Tuesday evening Dr. James B. Conant, President of Harvard University, will speak.

The laboratories of the Eastman Kodak Company will be open to visitors during the first and second days of the Symposium so that those who care to do so may see where and how Eastman Organic Chemicals are made, as well as inspect the photographic research laboratories of the Company. Arrangements can be made also for visits to other Rochester laboratories and industrial plants.

Many important contributions to science have been made at past symposia. Among the most outstanding are: the reports of Nieuwland on his work on acetylene; of Carothers on the polymerizations with which he has been so successful; of Kamm on his work on the active principles of the pituitary gland; and of Kendall on his researches on adrenaline; Conant's discussion of superacidity in acetic acid solution; Brooker's description of the preparation and use of dyes suitable for sensitizing photographic emulsions.

Rochester invites you to be present at the sixth biennial meeting.